424-411 AU 1502 49212

CA 002070231 A1
DEC 1992

93-059351/08 A92 C07 D23 SCEN- 91.06.04
SCENTRY INC *CA 2070231-A
91.06.04 91US-710121 (92.12.05) A61L 9/12, A01M 1/02, A01N 63/00

Volatile material release device with reduced burst effect - comprises polymeric container contg. cured matrix including active ingredient, used for insect pheromone(s), conserves expensive material, etc.

C93-026537

A(8-P1, 11-C2, 12-P1, 12-S10, 12-W4) C(4-C3B, 6-H, 7-H, 10-D1, 10-E4D, 10-G2, 10-J2, 12-L7, 12-M10A, 12-N3) D(10-A5B)

Release device for volatile material comprises a polymeric container having enclosed in it a cured matrix contg. an active ingredient capable of penetrating the matrix and container wall.

Also claimed is prepn. of the device by:
(a) mixing a polymeric resin and a plasticiser to form a plastisol matrix,
(b) mixing a volatile active ingredient into the matrix,
(c) placing the mixt. into a polymeric container, and
(d) curing the matrix.

USE/ADVANTAGE

The device provides a constant and regulated release rate over the entire surface area for an extended period of time, from 40 days to over 4 months. It has greatly reduced 'burst' effect, a rapid release in the first 1-3 days of active agent, with resultant wastage of a valuable chemical. Wastage is also avoided in mfr. of the device.

The device is intended partic. for release of behaviour modifying chemicals such as insect pheromones, which are extremely expensive, for monitoring and control of insects by mating disruption. The device can also be used for release of e.g. floral odours, perfumes and scents.

PREFERRED MATERIALS

The insect pheromone is an alkane, alkene alcohol, aldehyde or ester, epoxide or aromatic or heterocyclic cpd. The matrix is a cured plastisol of a polymeric resin esp. polyethylene or PVC and a plasticiser, esp. benzyl butyl phthalate (BBP).

PREFERRED MANUFACTURE

The device is prepd. using resin 40-60%, plasticiser 40-60% and agent 5-30%, all by wt. Other opt. addns., either to the matrix or the container are dye colourings and protective materials, e.g. against UV radiation.

The filled container is opt. wound round a rod before curing, to allow easy hanging. After curing, the device is cut to the required size.

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WIDER DISCLOSURE

The matrix contg. agent and container can also be mfd. by a co-extrusion process.

EXAMPLE

BBP (600 g) and PVC powder (400 g) were mixed to a homogeneous soln. and vacuumed to remove air bubbles. Tomato pinworm pheromone, Lycopersilure, (276 g) was added, mixed in, and vacuumed again. The mixt. was sucked into PVC tubing of internal dia. 0.125 inches, then plugged at both ends, wound round Al rods, and the ends taped. The rods were cured 6-12 mins at 250-300° F, then briefly immersed in cold water. The tubing was removed from the rods, cut into 1.577 g spirals, immediately packaged in foil bags.

The device released lycopersilure, after the first 2 days (9.5 and 5.2 mg/day) at a mean rate of 0.98 mg/day over a period > 4 months.(32pp2259CJGDwgNo0/5)

Addnl. Data: DOANE C C, JENKINS J W, WILSON W W
92.06.02 92CA-2070231

CA2070231-A

Consommation et Corporations Canada
Consumer and Corporate Affairs Canada

Bureau des brevets
Patent Office

Ottawa, Canada
K1A 0C9

(21) (A1) 2,070,231
(22) 1992/06/02
(43) 1992/12/05

(51) INTL.CL.[5] A61L-009/12; A01M-001/02; A01N-063/00

(19)(CA) **APPLICATION FOR CANADIAN PATENT** (12)

(54) Release Device for Volatile Materials and a Method of Making the Same

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(30) (US) 07/710,121 1991/06/04

(57) 20 Claims

Field of the Invention

The present invention relates to a controlled release device, in particular, a controlled release device for volatile materials and a method of making the same.

Background of the Invention

Insect sex pheromones are attractive chemical scents released in minute quantities by one sex to attract the other for mating. For example, many male moths can detect and follow minute quantities of pheromone carried by air currents, sometimes over long distances.

Researchers have identified over 600 different insect pheromone components. The natural pheromone released by the female may be composed of just one component while other species may release several or more different chemicals. After identification the pheromone components are chemically synthesized and suitably attractive mixes can then be formulated. Formulation is concerned with putting the pheromone into suitable controlled release devices so that they can be released at given rates over long periods of time. This is extremely important since several pheromones cost over $10,000 per kilo.

The two major uses for synthetic pheromones are in the monitoring and control of insect populations. Monitoring

involves the use of traps or other devices baited with pheromone in a controlled release device to attract and trap adult insects, usually the males. A common application is for monitoring for the presence and density of newly emerged adults so that appropriate control measures can be taken. Traps are also used for detection of new introduced pest species.

The second major use of insect pheromones is control of insects through mating disruption. Pheromone is loaded into slow-release dispensers that are then distributed evenly over wide areas. The males of the target insect species living within the treated area are unable to locate females, hence a measure of control is attained since unmated females do not lay fertile eggs. Several modes of action may be involved in mating disruption including adaptation/habituation of the males sensor system; false trail following, where males are preferentially attracted to, and follow pheromone plumes emanating from the release devices, and camouflage where the pheromone being released by the devices overpowers and camouflages the plumes from the live females.

The major requirement for monitoring and mating disruption is the controlled release of pheromone. To obtain suitable control it is necessary to release the pheromone at a constant level over several weeks or even months. The

pheromone must also be protected from external factors such as air, water and ultraviolet light so that it does not degrade.

There are several commercial release devices presently being developed or sold for mating disruption. The Scentry Celcon hollow fiber and the Scentry sprayable microencapsulated particle, the Hercon chip or trilaminate and the Mitsubishi rope are a few. Scentry and others have used polyvinyl chloride (PVC) release systems in the past. These are commonly prepared by mixing the pheromone in a plastisol and then exposing the plastisol to heat to cure and harden it. Scentry and others have used dip molding, pan molds, extrusions of PVC with heat to make dispensers. A major defect of these formulations is that a significant amount of pheromone may be lost during the heating or curing process. These techniques tend to be both wasteful and inaccurate since loss of pheromone may vary from batch to batch or product to product. Loss of pheromone during this process also effects the active ingredient specifications. It is critical to deliver the specified amount of technical pheromone in each dispenser since it is unlawful to deliver less than label rates.

The need for effective release devices for volatile materials, in particular pheromones, which release an active

ingredient at a constant rate, tend to be in great demand. Since some active ingredients, such as pheromones, may cost from $1,500.00 up to $12,000.00 per kilo, there is a demand for release devices which are less expensive to produce and are more effective in releasing a volatile material at a constant rate.

The production of present release devices can be exemplified as follows, the active ingredient is generally placed into a molten substrate, e.g., polymer contained in a heated vessel. A mandrel is heated in a furnace, then on exiting the furnace is lowered into the molten substrate which adheres to the "fingers". The mandrel is raised clear of the molten pheromone containing substrate and the adhering polymer cured by passage through a second furnace. Air cooling follows and the pheromone containing devices are separated from the mandrel by blasts of compressed air. However, during such a procedure, there exists the problem of the active ingredient vaporizing and releasing from the molten substrate and during curing. Industry estimates place losses of the active ingredient at 26% of the total amount initially in the molten substrate. Obviously, such losses are economically impractical and greatly increase production and hence product costs.

With existing methods of producing release devices for volatile materials, there tends to be no way to accurately

formulate dispensers for monitoring lures or for registered products for control of pests in the field. Also, with present release devices, many pheromones contained therein degrade quickly when exposed to ultraviolet light or oxygen.

A further problem in the industry is known as the "burst effect." The "burst effect" occurs when the active ingredient is released initially at a considerably higher than desired rate. In some cases, the "burst effect" can release 20% of the active ingredient in the first 1-3 days.

U.S. Patent No. 4,639,393 is said to disclose dispensers for the controlled release of pest-controlling agents, wherein laminated articles are provided which comprise one or more layers containing active pest control and pest attractant substances and which allow the controlled release of the pest controlling substance from within the laminate to the surface of the laminate.

U.S. Patent No. 4,445,641 is said to disclose a controlled-release dispenser which comprises a rate-controlling membrane and a solid microporous polymeric reservoir having interconnected or continuous pores capable of retaining the active ingredient by capillary forces.

U.K. Patent Application No. GB 2 141 932 A is said to disclose compositions for pest control which comprises a pheromone or a pheromone inhibitor in liquid or semi-liquid

form, in a polymeric water-resistant matrix which provides UV protection and which allows an adequate rate of diffusion of the active material over a prolonged period of time. The composition is then applied in the form of large globules or spots.

European Patent Application No. 0 243 007 is said to disclose a tubular sustained-release dispenser of insect sex pheromones wherein the pheromone permeates through a barrier wall made of a polymeric material.

European Patent Application No. 0 233 109 is said to disclose a tubular plastic vapor dispenser for sustained releasing vapor of a sex pheromone which has a deformed configuration forming at least one loop.

U.S. Patent No. 4,600,146 is said to disclose a vapor-releasing body of an elongated form capable of sustainedly releasing vapor of a vaporizable substance such as a sex pheromone. The integrated structure is composed of a capillary tubing of a polymeric material filled with the vaporizable substance.

The article by Weatherston, "Alternative Dispensers for Trapping and Disruption", Insect Pheromones in Plant Protection, 1989, discloses a method for preparation by mixing a resin and plasticizer together (with the addition of an antioxidant) and removing air bubbles by vacuum

evacuation, wherein a pheromone is then added and air bubbles removed again by vacuum evaporation. The mixture is then placed in a glass tube and the mixture fused at 145°C for 2-5 minutes or until it becomes translucent. The mold is then broken and the plastic rod slid out and cut into the desired lengths. Fitzgerald et al., "Slow Release Plastic Formulation of the Cabbage Looper Pheromone cis-7-Dodecenyl Acetate: Release Rate and Biological Activity" discloses a similar method of preparation.

None of the above dispensers (and methods of making the dispensers) overcome all of the problems described above.

Thus, there is a need for a more stable release device for volatile materials and a method of making the same which overcome the problems and disadvantages described above.

SUMMARY OF THE INVENTION

One object of the present invention is to provide a release device which provides a constant and regulated rate of release over the entire surface area of the device for an extended period of time.

Another object is to provide a release device which can be configured so as to be used both as a monitoring lure and also as a disruption formulation for direct control of pest species when pheromone is used as an active ingredient.

A further object is to provide a release device which greatly reduces the "burst effect".

A still furtner object is to provide a method of making a release device which greatly conserves the active ingredient from loss during the preparation process.

An additional object is to provide a method of making a release device which allows accurate formulation of the volatile material in the release device.

Additional objects and advantages of the invention will be set forth in part in the description which follows, and in part will be obvious from the description, or may be learned by practice of the invention. The objects and advantages of the invention will be realized and attained by means of the elements and combinations particularly pointed out in the appended claims.

To achieve the objects and in accordance with the purpose of the invention, as embodied and broadly described herein, the present invention is directed to a controlled release device for a volatile material comprising a polymeric container having enclosed therein a cured matrix containing an active ingredient capable of permeating the matrix and the wall of the container.

The present invention is also directed to a method of making a controlled release device for a volatile material

comprising mixing a volatile active ingredient with a polymeric resin and placing the mixture into a polymeric container.

The present invention is further directed to a method of making a controlled release device for a volatile material comprising mixing a polymeric resin and a plasticizer to form a plastisol matrix; mixing a volatile active ingredient into the plastisol matrix; placing the resultant mixture into a polymeric container; and then curing the plastisol matrix containing the volatile active ingredient.

It is to be understood that both the foregoing general description and the following detailed description are exemplary and explanatory only and are not restrictive of the present invention, as claimed.

BRIEF DESCRIPTION OF THE DRAWINGS

Figures 1 and 2 are graphs which illustrate the release rates from release devices of the present invention.

Figures 3, 4 and 5 are graphs which illustrate evaluations of comparisons of the reduction in trap catch of male tomato pinworm moths using the device of the present invention with male trap catch in check fields treated conventionally with insecticides.

DETAILED DESCRIPTION OF THE INVENTION

The present invention is a device for releasing, over an extended period of time and at a constant rate, volatile materials including, but not limited to, insect pheromones and other behavior modifying chemicals. The device of the present invention can be used in both the monitoring and direct control of pest insects by dissemination of synthetic pheromones within a crop area to either attract one or both sexes of the pest species to a trap; or to disrupt the sexual communications between the male and female insects thereby reducing mating and causing the deposition of unfertilized eggs with a concomitant reduction in the subsequent generation.

The release device for volatile materials comprises a polymeric container having enclosed therein a cured matrix containing an active ingredient capable of permeating the matrix and the wall of said container.

Any polymeric container for use in the present invention is acceptable. In one embodiment, the polymeric container is a tube made from polyvinylchloride or polyethylene and is of food grade. One commercially available polymeric tube is from Rehau Plastics in Leesburg, Virginia. Other suitable polymeric containers can be made from for example, other polyolefins such as polypropylene; copolymers of ethylene and

vinyl acetate; cellulose acetate; formalized polyvinyl alcohol; polymers of acrylic and methacrylic esters; polyamide resins; polyesters; and polyorganosiloxanes.

The internal diameter of the polymeric tube can range from about 30 mil. to about 0.5 inch with a wall thickness in the range of about 0.016 inch to about 0.25 inch.

The polymeric or plastisol matrix which will contain the active ingredient is prepared by the polymerization of a mixture of a polymeric resin with a plasticizer, e.g., of a plastisol. One typical example of a polymeric resin used in the present invention is Geon 138 polyvinylchloride obtainable from Goodrich Inc., Cleveland, Ohio and one typical example of a plasticizer is benzyl butyl phthalate obtainable from Monsanto, St. Louis, Missouri. Other suitable polymeric resins include, for example, esters of acrylic acid, esters of methacrylic acid, vinyl acetate, vinyl pyrrolidone and styrene. Other suitable plasticizers include, for example, other organic esters such as terephthlates, trimellitates, adipates, sebacates, etc.

The range percentage for the polymeric resin can vary, but preferably, is from about 40-60% by weight of the total polymeric matrix including the active ingredient. The range percentage for the plasticizer can also vary, but is preferably about 40-60% by weight of the total polymeric matrix including the active ingredient.

Any type of volatile material (active ingredient) which is desired to be released over an extended period of time and at a constant rate can be used with the device of the present invention, e.g., floral odors, perfumes, scents. In particular, behavior modifying chemicals are used as the active ingredient, i.e., pheromones, kairomones, etc. Specific examples of pheromones include, but are not limited to, alkanes (e.g., methylnonadecane), alkene aldehydes (e.g., Z-11-hexadecenal), alkene alcohols (e.g., E,E-8-10-dodecadien-1-ol), epoxides (e.g., disparlure), alkene esters (e.g., gossyplure) and aldehydes derived therefrom, aromatic compounds (e.g., benzaldehyde) and heterocyclic compounds (e.g., Japonilure). The pheromones disclosed, for example, in U.S. Patent No. 4,734,281 and incorporated herein by reference may be also used. One specific example of a pheromone is the tomato pinworm pheromone which is sold under the tradename LYCOPERSILURE.

The range percentage for the active ingredient can also vary, but preferably is from about 5-30% by weight of the total polymeric matrix including the active ingredient.

Optionally, a dye (e.g., organic pigments such as pigment scarlet, diarylide orange, indanthrone and carbon black; soluble dyes such as aniline black and anthraquinone; and inorganic pigments such as titanium dioxide, chrome, iron

oxide and chromium oxide - all commercially available) can be added to the polymeric container or to the polymeric matrix at the time of mixing the polymeric resin and plasticizer, afterwards, or with the addition of the active ingredient.

The amount of dye added can range from about 0.1% to 2.0% by weight of the release device.

Also, protective materials, such as UV protectants and antioxidants, can be added to the polymeric container or to the polymeric matrix at the time of mixing the polymeric resin and plasticizer, afterwards, or with the addition of the active ingredient. Typical examples of UV protectants include Advastab BC109, Carstab and any of the class of compounds known as hindered benzophenones. Typical examples of antioxidants include Sustane, Tenox, Butylated Hydrox-Toluene, Butylated Hydroxy Anisole.

The amount of the protective material added can range from about 0.01% to 1.0% by weight of the polymeric matrix with active ingredient.

In one method for making the controlled release device for volatile materials, the method comprises the following steps:

(1) mixing a polymeric resin and a plasticizer to form a plastisol matrix;

(2) mixing a volatile active ingredient into said plastisol matrix;

(3) placing the resultant mixture into a polymeric container; and

(4) curing the plastisol matrix containing the volatile active ingredient.

In preparing the matrix, which is preferably polymeric, a plasticizer is added to a polymeric resin, such as a PVC powder, and the mixture is stirred with, for example, an electric stirrer, for approximately 30 minutes until a homogeneous solution is obtained. The resulting plastisol is preferably placed under vacuum or other suitable means for a time sufficient, e.g., approximately 30 minutes, to remove all air bubbles.

The active ingredient is then added to the plastisol and the resulting mixture is stirred (as above) for approximately 30 minutes and preferably vacuumed again for a time sufficient, e.g., 30 minutes, to remove any existing air bubbles.

The resulting mixture, which is the plastisol containing the active ingredient, is then filled into a container, e.g., a polymeric tube. Any suitable filling technique may be used, preferably vacuum suction. The filled tubing is then preferably plugged at both ends with plugs. While any type

of plug may be used, plastic irrigation plugs are preferred. The filled tubing is then heated at a time and temperature sufficient to cure the plastisol and form the matrix which also bonds to the tubing, e.g., time: 6 to 12 minutes and temperature: 250°F to 300°F.

Besides using heat to cure the plastisol containing the volatile active ingredient, any other known means may be used to cure the plastisol containing the volatile active ingredient.

In one embodiment of the present invention, the filled tubing can be firmly, but not tightly, wound around rods (e.g., aluminum rods), wherein the ends of the wound, filled tubing are taped down leaving enough of the rod ends exposed to fit into the racks of an oven. The prepared rods are then heated in the oven for approximately 6-12 minutes at 250-300°F.

Of course, the filled tubing prior to heating does not have to be wound around rods, but can simply be placed in an elongated shape in the heat for the same amount of time.

At the end of the curing period, the filled tubing is removed from the oven and preferably briefly immersed in cold water. At this point, the polymeric matrix containing the active ingredient inside the polymer tubing will now be solid and clear and completely bonded with the polymeric tubing

along its whole length, thus stabilizing and regulating the release of the active ingredient over the whole surface area of the device.

The tubing once removed from the water (if used), then from the rods, if used, can be cut to desired lengths with a razor blade or any other cutting device, preferably into approximately 3.25 inch spirals which weigh approximately 1.577 grams, and immediately packaged in foil-lined bags and stored at approximately 45-50°F.

If rods are used in the curing step, the spiral design of the cured, filled tubing allows the release device to be configured in such a way so as to allow easy hanging of the device in desired locations.

Another method of making the controlled release device involves a coextrusion process. In one such process, a plastic extruder is used. In this process, plastic powder or granules of the polymeric resin (e.g., PVC) are converted into continuous uniform melts and forced through a die to yield a desired shape (e.g., tube). The coextrusion dies used are generally of a nested configuration (e.g., a tube of smaller diameter inside a tube of larger diameter), wherein individual polymers flow through separate mandrel passages (e.g., each separate tube). Thus, in this process the matrix described above will be formed in an inner section of the

coextrusion die and the polymeric container will be formed in an outer section of the coextrusion die. Prior to entering the inner section of the die, the volatile active ingredient is injected into the matrix, preferably adjacent the die tip. The amounts of the active ingredient to be used would be the same as described above. Once exiting the die, the container and matrix will bond together to form the controlled release device and preferably the device then enters a cooling chamber to harden the resulting device. (Preferably, the device has the same range of dimensions as described above.) Once the controlled release device is formed and hardened, it may be cut into any desired length. In determining which polymers or thermoplastic materials to use in this process, it is important that adjacent polymers exhibit similar flow and adhesion characteristics to form the coextrusion.

Also, dyes and/or protective materials as described above may be included in the device simply by injection into the desired section, prior to entering the die, i.e. - inject into the matrix extrusion or into the container extrusion. The amounts to be used would be the same as described above.

The present invention will be further clarified by the following examples, which are intended to be purely exemplary of the present invention.

EXAMPLE 1

600 g of benzyl butyl phthalate was added to 400 g of PVC powder and the mixture was stirred with an electric stirrer for 30 minutes until a homogeneous solution was obtained. The resulting plastisol was put under vacuum for 30 minutes to remove air bubbles. 276 g of Lycopersilure (the tomato pinworm pheromone) was added to the plastisol and the mixture was stirred and vacuumed again for 30 minutes. The mixture was then sucked into a PVC tubing [i.d. 0.125" o.d. 0.188"]. The filled tubing was plugged at both ends with plastic irrigation plugs. The filled tubing was then firmly, but not tightly, wound around aluminum rods [o.d. 0.5" by 17.25" long], the ends of the wound tubing were taped down with tape, for example, 3M 810, leaving enough of the rod ends exposed to fit the racks of the oven. The prepared rods were heated in the oven for 6-12 minutes at 250-300°F (actual oven temperature may reach 310°F). At the end of the curing period, the rods were removed from the oven and briefly immersed in cold water. The polymeric matrix (containing the pheromone) inside the PVC tubing was now solid and clear. The filled tubing was removed from the rods and cut, with a razor blade, into 3.25" spirals weighing 1.577 g and immediately packaged in foil-lined bags, 500 spirals to a bag, and stored at approximately 45-50°F.

Figures 1 and 2 illustrate the release rate of the release device constructed. The data indicate that the device releases the Lycopersilure at a mean rate of 0.98 mg/day (S.D. 1.58 mg/day) over a period in excess of 4 months.

EXAMPLE 2

Release devices made in accordance with Example 1 were used in field tests to determine the efficiency of the present invention.

In particular, the efficiency of the release devices was demonstrated in the summer of 1990 on fresh market tomatoes growing in Baja California, Mexico. These tests (three replicates) were carried out in cooperation with agronomists from Quimical. The evaluation of the spirals was measured by the reduction in trap catch of male tomato pinworm moths in fields treated with the release devices of the present invention compared to the male trap catch in check fields treated conventionally with insecticides.

Treatments were made at two rates, 200 devices/acre (LOW RATE) and 400 devices/acre (HIGH RATE). The results given in Figures 3, 4 and 5 clearly demonstrate the effectiveness of the device. In Figure 3, both the high and low rate were equally effective in disrupting the mating communication of

the pest species as evidenced by the dramatic reduction in trap catch over that from the check field. The formulation proved to be efficacious for a period in excess of 40 days at which time the test was terminated. In the second replicate (Figure 4), both the high and low rates again performed equally well, being efficacious for a period in excess of 110 days.

In the third replicate (Figure 5), both rates performed excellently as compared to the check over the first 30 days, at which time the check field was also treated with the devices. Subsequently, the population of the moths in all three fields were equally suppressed.

Since virtually all of the active ingredient that is metered into the plastisol is retained in the cured matrix, the preparation of this release device conserves all of the active ingredient so formulated.

In addition, the release device of the present invention can be configured so as to be used both as a monitoring lure and also as a disruption formulation for direct control of pest species. Other release devices, in particular pheromone dispensers, are not so versatile.

In addition, the release device of the present invention, whether used for monitoring or direct control,

exhibits a highly controlled and constant release rate as opposed to other conventional release dispensers which exhibit a very rapid release of an active ingredient, such as pheromone, the "burst effect" which unnecessarily releases initially a large amount of active ingredient.

Other embodiments of the present invention will be apparent to those skilled in the art from consideration of the specification and practice of the present invention disclosed herein. It is intended that the specification and examples be considered as exemplary only, with a true scope and spirit of the present invention being indicated by the following claims.

WHAT IS CLAIMED IS:

1. A release device for a volatile material comprising a polymeric container having enclosed therein a cured matrix containing an active ingredient capable of permeating said matrix and the wall of said container.

2. The release device according to claim 1, wherein said matrix is polymeric.

3. The release device according to claim 1, wherein said container is tubular.

4. The release device according to claim 1, wherein said matrix is a cured plastisol of a polymeric resin and a plasticizer.

5. The release device according to claim 4, wherein said polymeric resin is polyethylene or polyvinylchloride.

6. The release device according to claim 4, wherein said plasticizer is benzyl butyl phthalate.

7. The release device according to claim 1, wherein said active ingredient is a behavior modifying chemical.

8. The release device according to claim 7, wherein said behavior modifying chemical is an insect pheromone.

9. A method of making a release device for a volatile material comprising:

mixing a volatile active ingredient with a polymeric resin; and

placing the mixture into a polymeric container.

10. The method according to claim 9, wherein said polymeric resin is polyethylene or polyvinylchloride.

11. A method of making a release device for a volatile material comprising:

mixing a polymeric resin and a plasticizer to form a plastisol matrix;

mixing a volatile active ingredient into said plastisol matrix;

placing the resultant mixture into a polymeric container; and

curing the plastisol matrix containing said volatile active ingredient.

12. The method of claim 11, wherein said polymeric resin is polyvinylchloride or polyethylene and said plasticizer is benzyl butyl phthalate.

13. The method according to claim 11, wherein said polymeric resin is present in the amount of about 40-60%, said plasticizer is present in the amount of about 40-60%, and said volatile active ingredient is present in the amount of about 5-30%, all by weight of total composition.

14. The method according to claim 11, wherein said volatile active ingredient is a behavior modifying chemical.

15. The method according to claim 14, wherein said behavior modifying chemical is an insect pheromone.

16. The method according to claim 15, wherein said insect pheromone is selected from the group consisting of alkanes, alkene aldehydes, alkene alcohols, epoxides, alkene

esters and aldehydes derived therefrom, aromatic compounds and heterocyclic compounds.

17. The method according to claim 11, further comprising the step of adding a dye to said plastisol or to polymeric container.

18. The method according to claim 11, further comprising the step of adding a protective material to said plastisol or to said polymeric container.

19. The method according to claim 11, wherein said polymeric container is wound around a rod prior to said curing.

20. The method according to claim 11, further comprising the step of cutting the filled polymeric container to a desired length after curing.

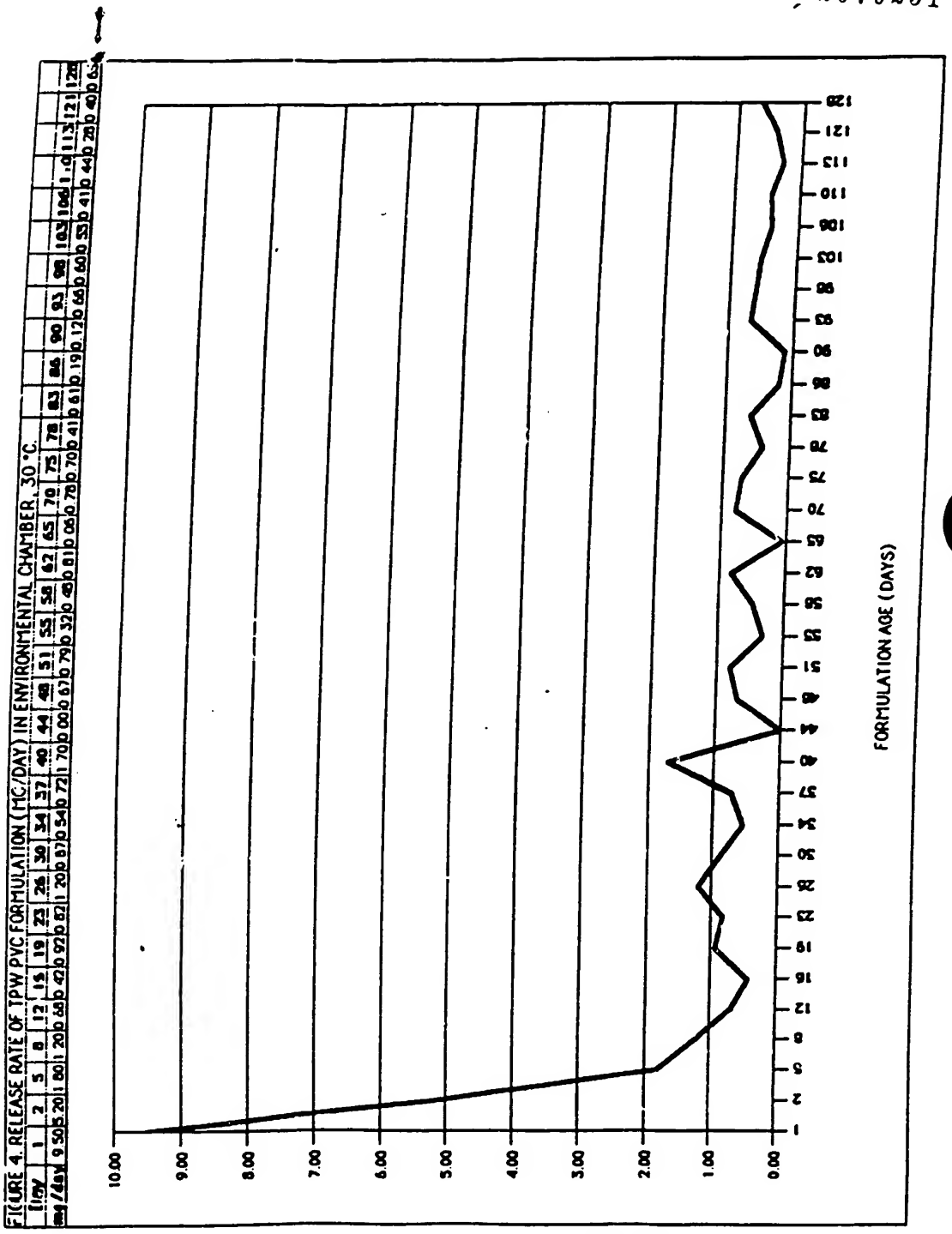
FIGURE 4. RELEASE RATE OF TPW PVC FORMULATION (MC/DAY) IN ENVIRONMENTAL CHAMBER, 30 °C.
FORMULATION AGE (DAYS)
10.00
9.00
8.00
7.00
6.00
5.00
4.00
3.00
2.00
1.00
0.00

FIGURE 1

FIGURE 5. RELEASE RATE OF TPW PVC FORMULATION (MG/DAY) IN ENVIRONMENTAL CHAMBER, 30 °C.

Day	1	2	5	6	7	8	9	12	14	16	20	24	27	31	36	41	44
mg/day	9.9	2.1	2.5	1.7	2.2	1.3	1.2	1.3	1.2	1.0	1.1	0.9	1.5	0.9	0.9	0.8	0.9

Day	48	51	55	58	61	65	68	72	76	79	84	88	93	96	100	103
mg/day	0.8	0.7	0.7	0.7	0.4	1.1	0.5	0.7	0.7	0.8	0.7	0.2	0.3	0.4	0.6	0.1

Day	107	111	114	117	121	128	132	136	142	146	149	153	156	162	169	176
mg/day	0.4	0.1	0.4	0.2	0.1	0.5	0.0	0.1	0.2	0.2	0.3	0.1	0.1	0.1	0.2	0.0

FIGURE 5. RELEASE RATE OF TPW PVC FORMULATION (MG/DAY) IN ENVIRONMENTAL CHAMBER, 30 °C.

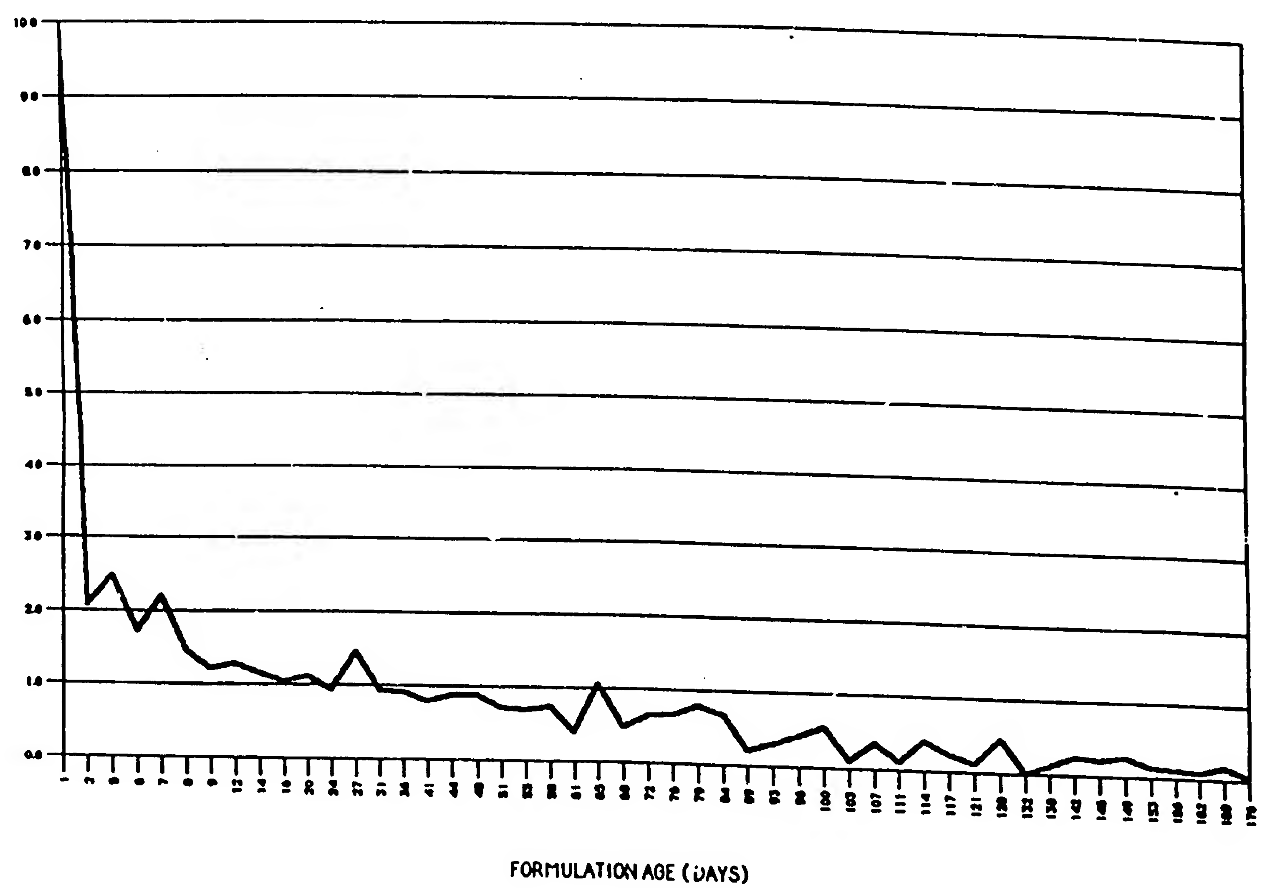

FIGURE 2

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ABSTRACT OF THE DISCLOSURE

A release device for volatile materials comprising a polymeric container having enclosed therein a cured matrix containing an active ingredient capable of permeating the matrix and the wall of the container and a method of making the same are disclosed.

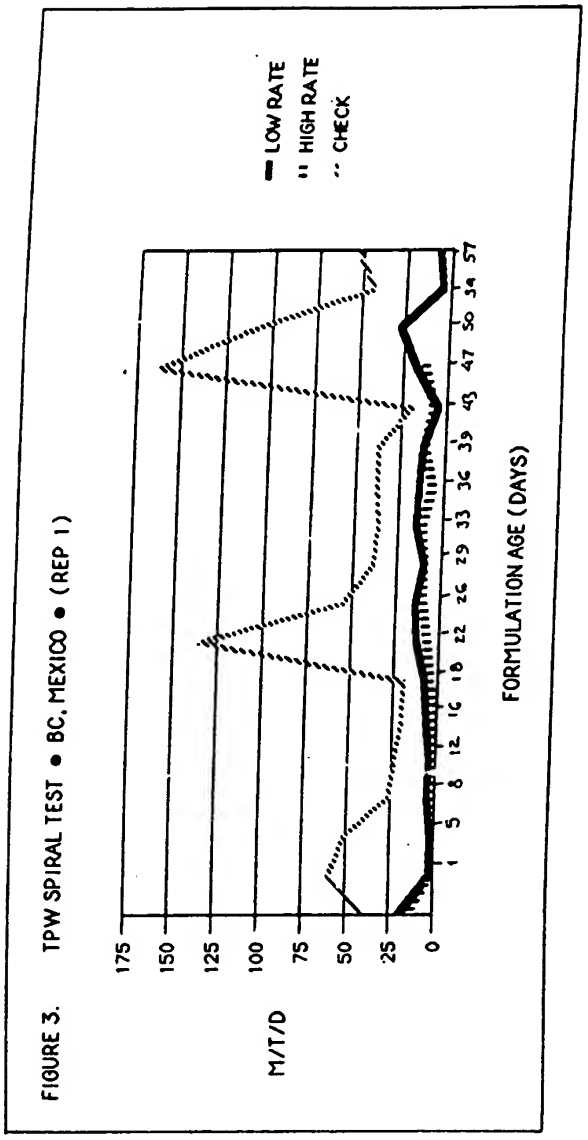

FIGURE 3. TPW SPIRAL TEST • BC, MEXICO • (REP 1)

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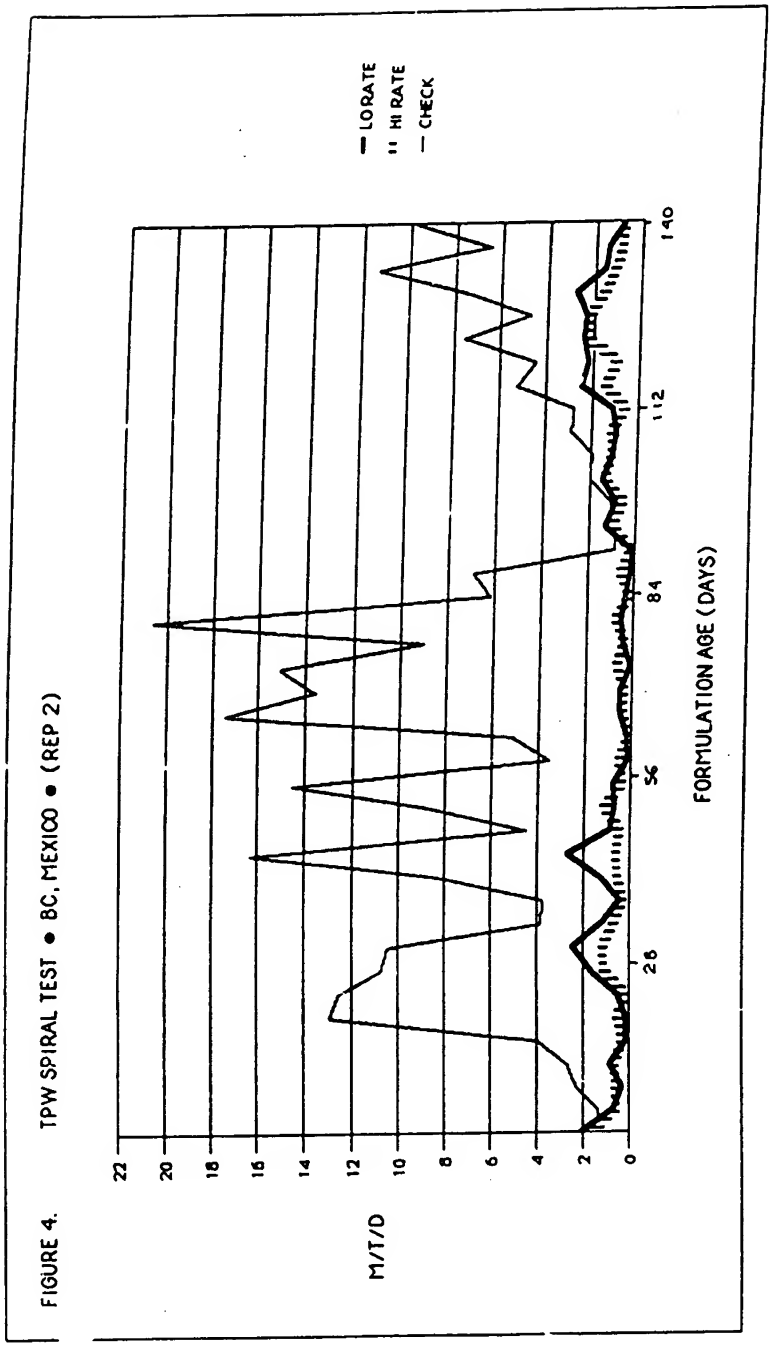

FIGURE 4. TPW SPIRAL TEST • BC, MEXICO • (REP 2)

Richard McKenzie & Hurlbut

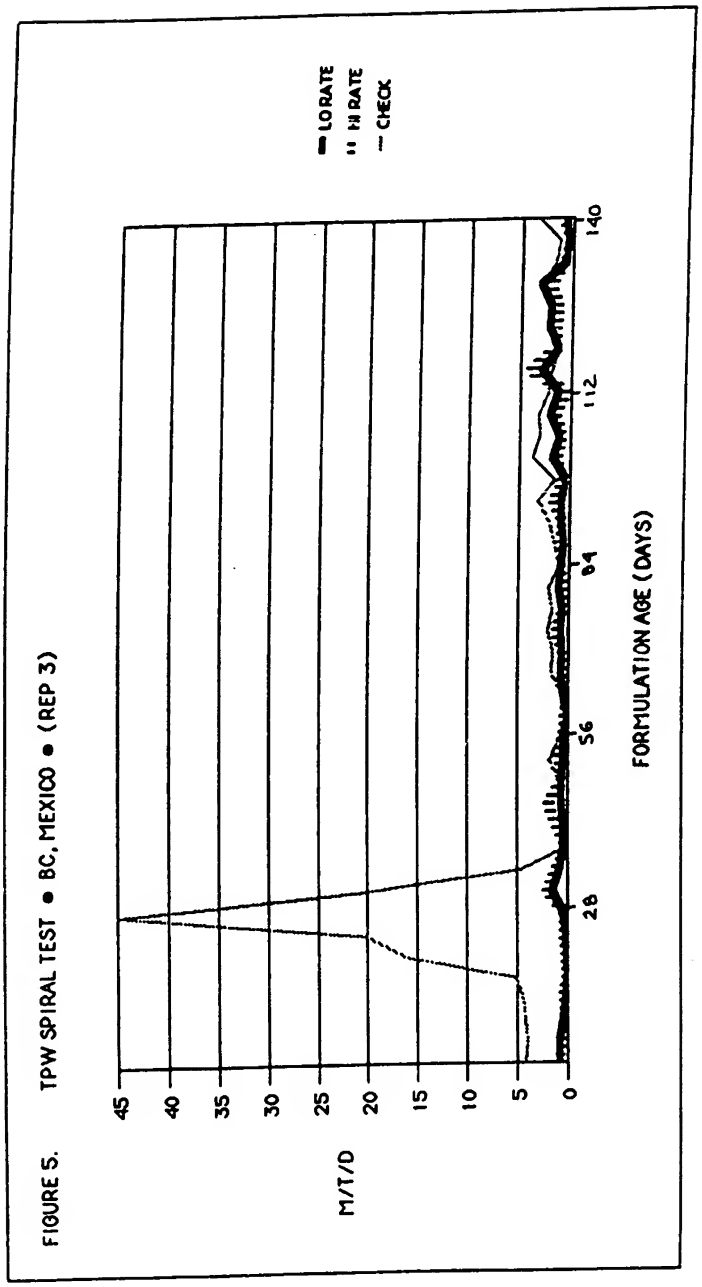

FIGURE 5. TPW SPIRAL TEST • BC, MEXICO • (REP 3)

Kirkes McKenzie & Herbert